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SM-48461-Q2

QUARTERLY PROGRESS REPORT

PROGRAM TO DEVELOP AN INORGANIC SEPARATOR FOR A HIGH TEMPERATURE SILVER-ZINC BATTERY

by

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prepared for

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Solar and Chemical Power Branch
Daniel G. Soltis

MISSILE & SPACE SYSTEMS DIVISION
ASTROPOWER LABORATORY
Douglas Aircraft Company, Inc.
Newport Beach, California

FOREWORD

This report was prepared under Project Work Order Number 177091-01, and covers the period October 29, 1965 through January 29, 1966. The research, development and testing activities discussed herein are sponsored by Lewis Research Center, National Aeronautics and Space Administration, Cleveland, Ohio, under Contract NAS 3-7639.

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1.0 INTRODUCTION AND SUMMARY

During the reporting period, work has continued related to the objectives of NASA Program NAS 3-7639 "Program to Develop an Inorganic Separator for a High Temperature Silver-Zinc Battery". As in the first quarter of this program,[1] work during the second quarter has emphasized component fabrication, testing and evaluation as specified under Task II of the work statement. Work has also continued on Task I which is devoted to cell and component design.

Task I of this program, related to cell design, is nearly completed. The five ampere-hour (5 AH) cell design that has been selected is based on the experimental work carried out on components in Task II and consists of a grooved frame subassembly of separators and electrodes. It is then assembled in a cell case along with the cover and terminals. The cell case, cover and frames are made from polyphenylene oxide (PPO). Detail and assembly drawings of this design are shown in Figures 1 through 5. Molded PPO cases have been ordered and should be received by about March 1, 1966.

Task II, concerned with component fabrication and evaluation, is also on schedule. The best components produced thus far are described and applicable test results are presented. Except for the zinc electrodes, where additional work must be done to satisfactorily meet all program requirements, the cell components are being finalized for use in Task III multiplate cells. A number of test cells have completed 140 cycles, as required by the work statement, at 25°C and 100°C at 20 ma/cm^2. Tests at higher rates are under way to determine practical discharge and charging limits. Two multiplate cells have been given a preliminary cycle test at 25°C. These cells completed 1165 and 427 cycles, respectively, when they were disassembled for component analysis but do not, however, represent the best assembly of components. The results of this component analysis are being used for further guidance of Task II component development efforts.

Physical property and KOH compatibility tests have been completed on Celcon and PPO case materials. Based upon these tests, PPO has been selected as the case material for use in Task III of this program.

In general, the results obtained during the first two quarters of this program are favorable and the entire program is on schedule.

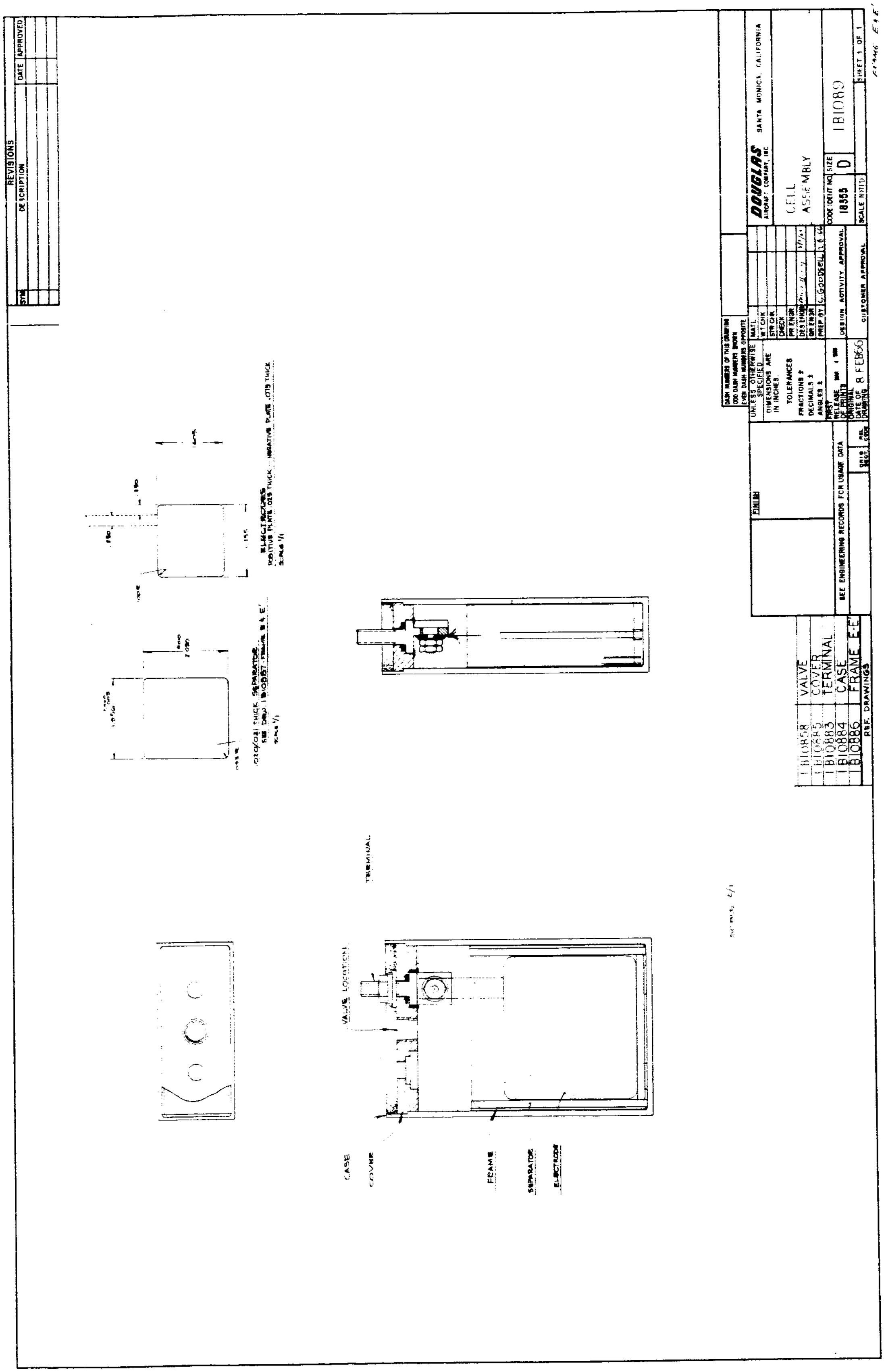


Figure 1. Cell Assembly

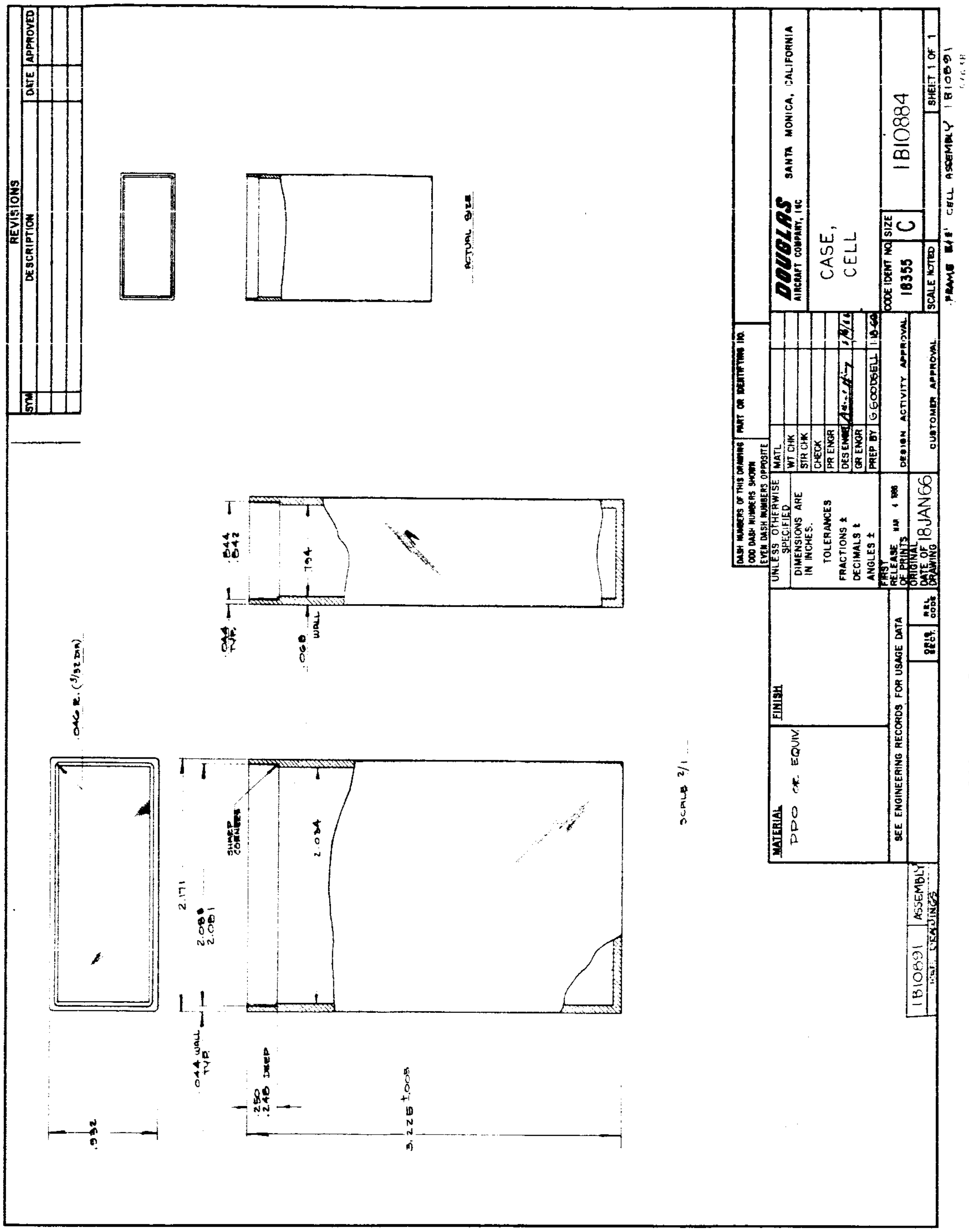


Figure 2A. Case, Cell

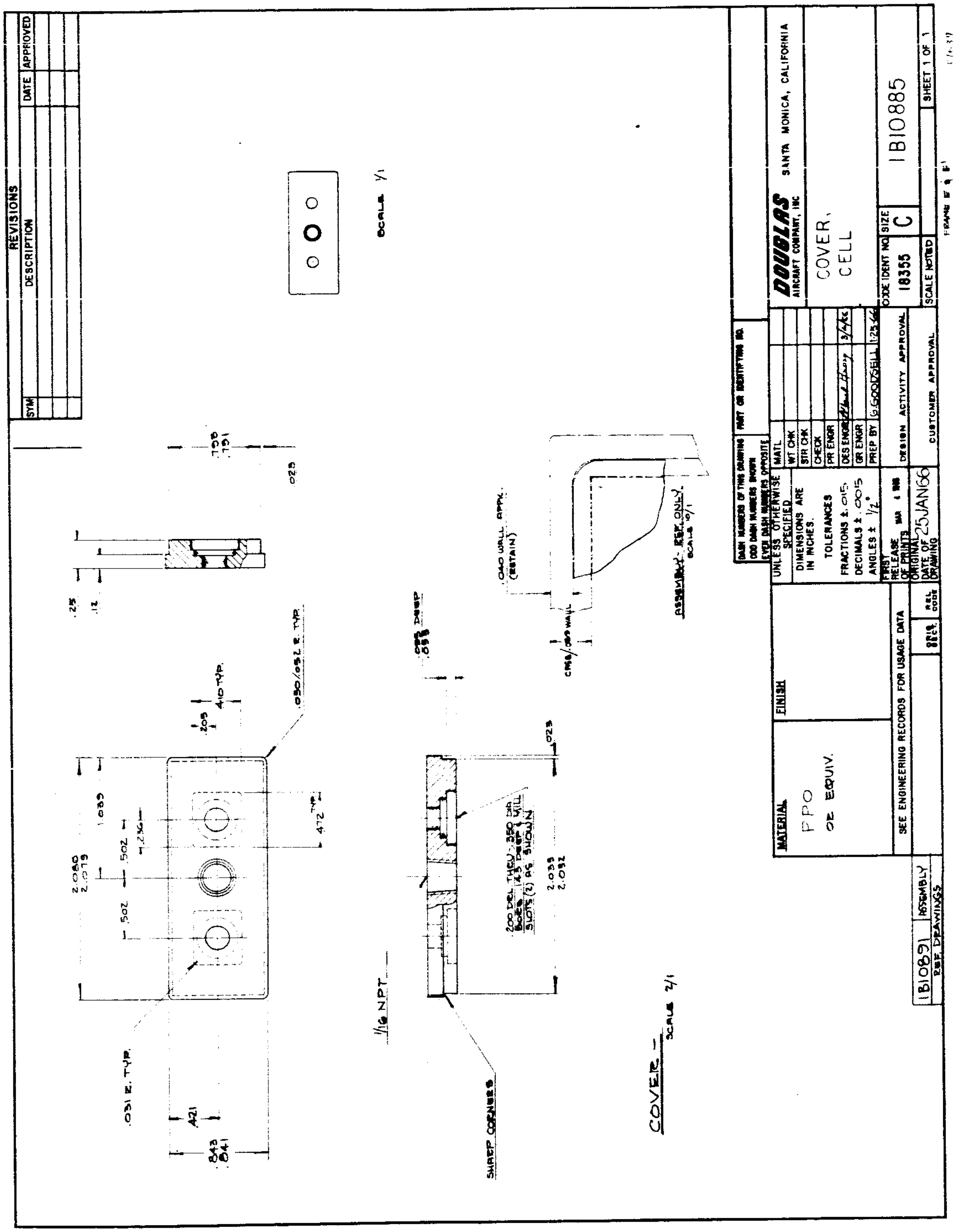


Figure 2B. Cover, Cell

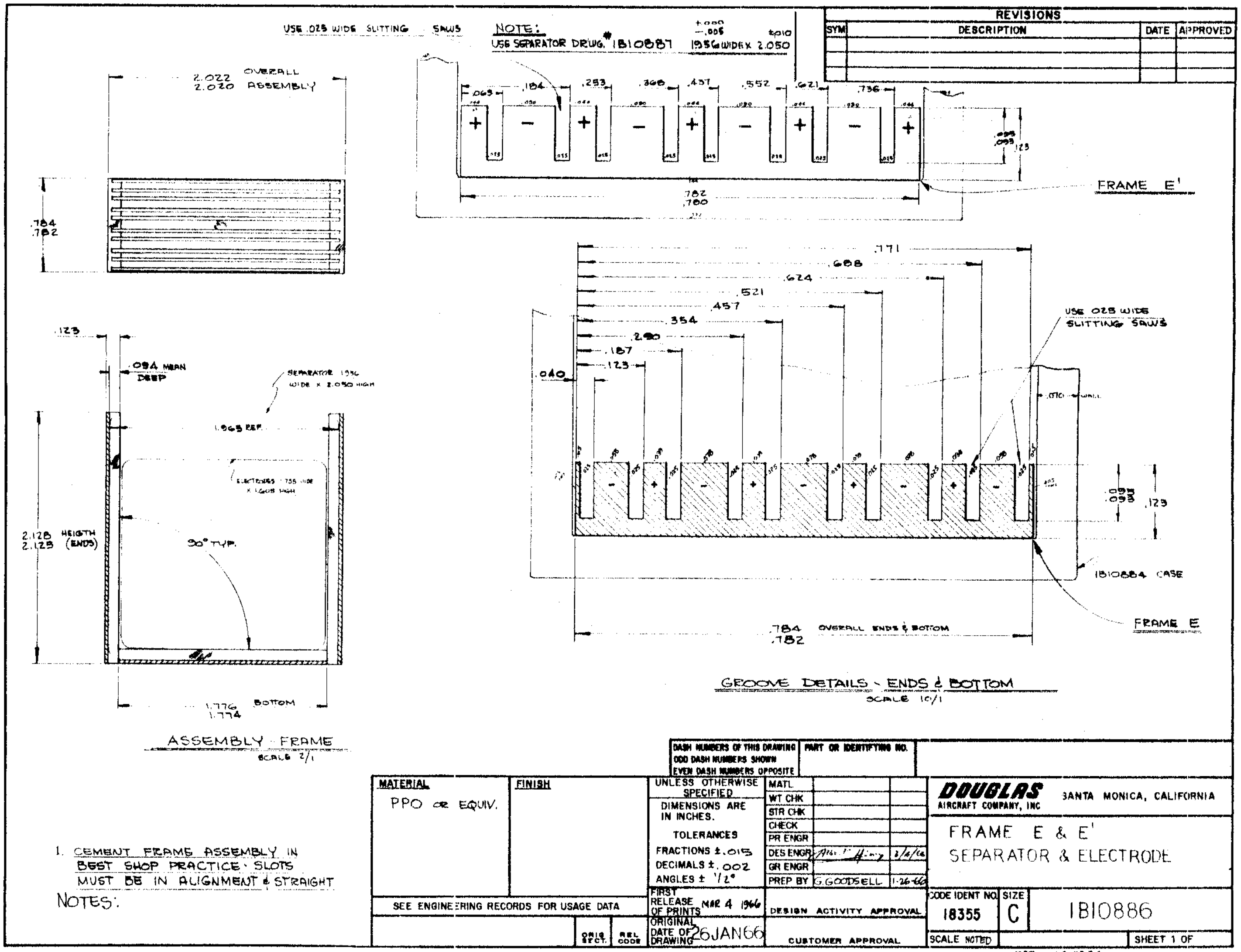
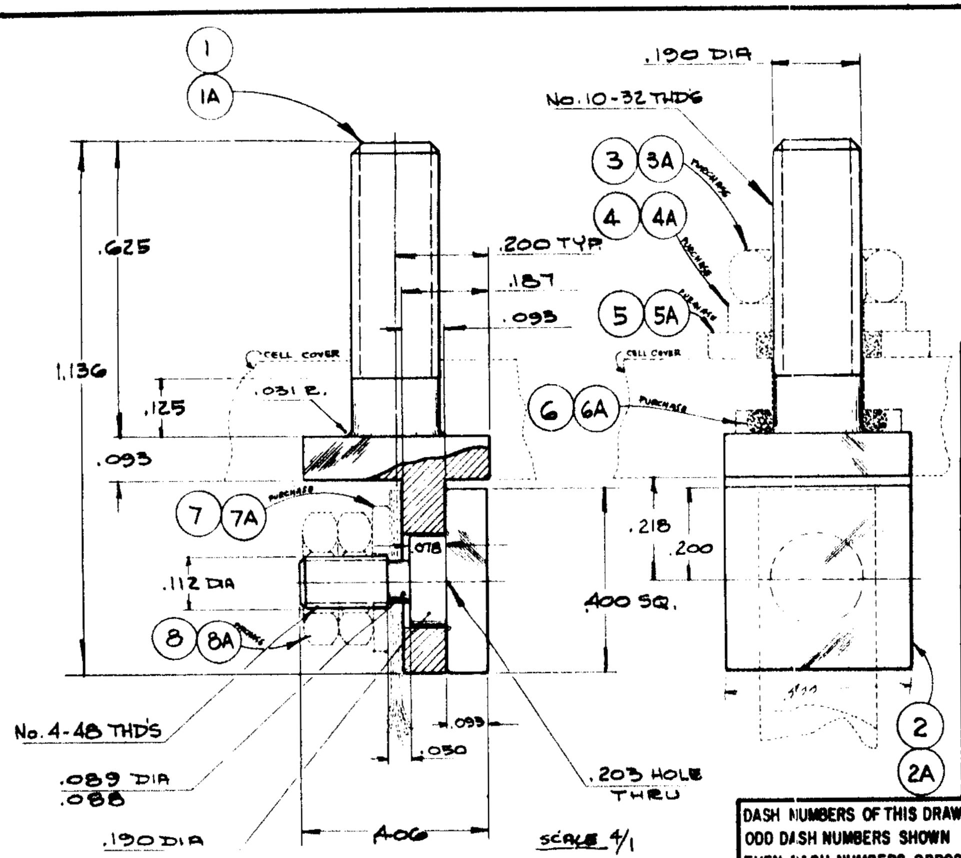
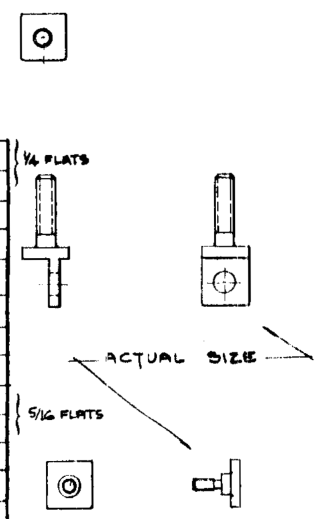


Figure 3. Frame E and E', Separator and Electrode



REVISIONS

SYM	DESCRIPTION	DATE	APPROVED



DET.	REQ	SIZE / DESCRIPTION / (PER CELL)	FINISH
8A	2	#4-48 HEX NUT 5/64 THK STAINLESS STEEL	SILVER PLATED
8	2	#4-48 HEX NUT 5/64 THK STAINLESS STEEL	NICKEL PLATED
7A	1	WASHER .031 THK 5/16 O.D. 1/8 I.D. STAINLESS STL	SILVER PLATED
7	1	WASHER .031 THK 5/16 O.D. 1/8 I.D. STAINLESS STL	NICKEL PLATED
6	2	"O" RING - PARKER # 2-8 E 515-8 CAL-STATE SEAL CO.	GARDEN GROVE
5	2	THREAD SEALS - PARKER # 7500-10 (EP INSERT)	CAL-STATE SEAL
4A	1	WASHER .062 THK. 3/8 O.D. 13/64 I.D. STAINLESS	SILVER PLATED
4	1	WASHER .062 THK. 3/8 O.D. 13/64 I.D. STAINLESS	NICKEL PLATED
3A	1	#10-32 HEX NUT 7/64 THK STAINLESS	SILVER PLATED
3	1	#10-32 HEX NUT 7/64 THK STAINLESS	NICKEL PLATED
2A	1	.400 SQ X .406 STAINLESS STEEL	SILVER PLATED
2	1	.400 SQ X .406 NICKEL	
1A	1	.400 SQ X 1.136 LG STAINLESS STEEL	SILVER PLATED
1	1	.400 SQ X 1.136 LG NICKEL	

DASH NUMBERS OF THIS DRAWING
ODD DASH NUMBERS SHOWN
EVEN DASH NUMBERS OPPOSITE

PART OR IDENTIFYING NO.

MATERIAL	FINISH
NOTED	NOTED

SEE ENGINEERING RECORDS FOR USAGE DATA

ORIG SECT. | REL CODE

UNLESS OTHERWISE SPECIFIED
DIMENSIONS ARE IN INCHES.
TOLERANCES
FRACTIONS ± .015
DECIMALS ± .005
ANGLES ± 1/2°

FIRST RELEASE OF PRINTS 14 JAN 66
ORIGINAL DATE OF DRAWING 14 JAN 66

MATL		
WT CHK		
STR CHK		
CHECK		
PR ENGR		
DES ENGR	[signature]	1/14/66
GR ENGR		
PREP BY	G. GOODSELL	1-14-66

DESIGN ACTIVITY APPROVAL

CUSTOMER APPROVAL

DOUGLAS AIRCRAFT COMPANY, INC — SANTA MONICA, CALIFORNIA

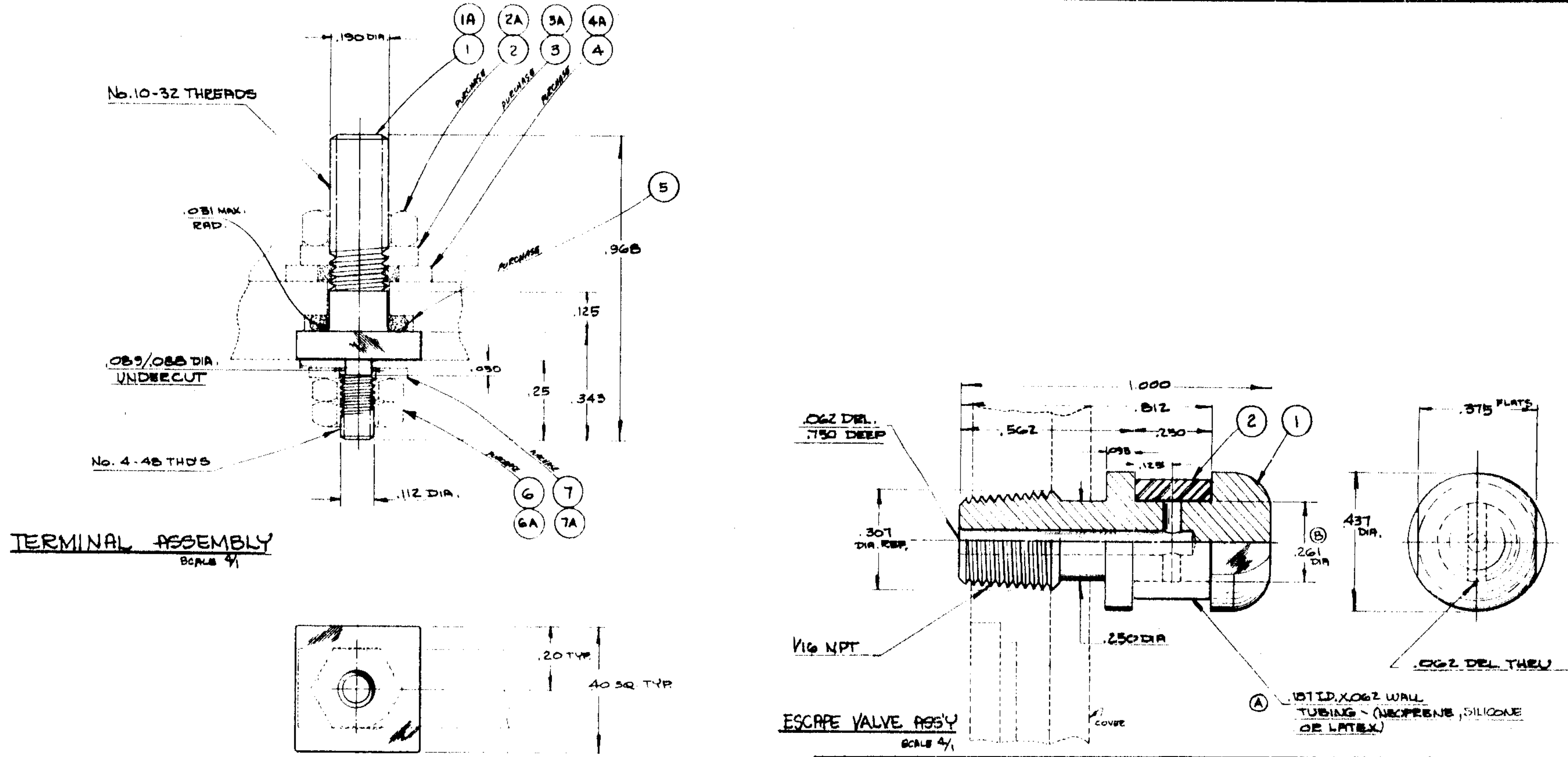
Figure 4.
Assembly, Cell Terminal

CODE IDENT NO.	SIZE	
18355	B	1B10883

SCALE NOTED — SHEET 1 OF 1

REF. DRWG. 1B10859 ASSEMBLY

REVISIONS				
SYM	DESCRIPTION		DATE	APPROVED
A	SILICONE MATERIAL ADDED	ℌ	1-8-66	
B	WAS .250 DIA.	ℌ	2-18-66	



MATL. NOTES —

TERMINAL ASS'Y DETAILS - PER CELL

(1A)(1) TERMINAL POST (1) STAINLESS STL. (1) NICKEL (2) REQ'D
(2A)(2) NUT - HEX #10-32, 7/64 THK, 5/16 ACROSS FLATS (2) REQ'D
(3A)(3) WASHER - .062 THK, 3/8 O.D., 13/64 HOLE (2) REQ'D
(4A)(4) THREAD-SEAL - PARKER #7500-10 (2) REQ'D
(5) "O" RING - PARKER #2-8 E 815-8 (2) REQ'D
(6A)(6) NUTS - HEX #4-48, 5/64 THK, 1/4 ACROSS FLATS (4) REQ'D
(7A)(7) WASHER - .031 THK, 5/16 O.D., 1/8 HOLE (2) REQ'D

ESCAPE VALVE DETAILS

(1) VALVE - STAINLESS STEEL (1) REQ'D
(2) TUBING - .187 I.D., .062 WALL, .250 LONG (1) REQ'D

LIST OF MATERIALS

PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	CODE IDENT NO.	STOCK SIZE	MATERIAL DESCRIPTION	MATERIAL SPECIFICATION	FIND NO.	ZONE

DASH NUMBERS OF THIS DRAWING
ODD DASH NUMBERS SHOWN
EVEN DASH NUMBERS OPPOSITE

QUANTITY REQUIRED PER NOTED ASSY

MATERIAL: NOTED

FINISH
DETS. 2,3,4,6,7 NICKEL PLATED STAINLESS STEEL
DETS. 1A, 2A, 3A, 4A, 6A, 7A SILVER PLATED STAINLESS STEEL

SEE ENGINEERING RECORDS FOR USAGE DATA

ORIG. SECT. | REL. CODE

UNLESS OTHERWISE SPECIFIED
DIMENSIONS ARE IN INCHES.
TOLERANCES
FRACTIONS ±
DECIMALS ±
ANGLES ±

MATL		
WT CHK		
STR CHK		
CHECK		
PR ENGR	[signature]	12/8/65
DES ENGR		
GR ENGR		
PREP BY	G. GOODSELL	12-6-65

FIRST RELEASE OF PRINTS 7 DEC 65
ORIGINAL DATE OF DRAWING 7 DEC 65
DESIGN ACTIVITY APPROVAL
CUSTOMER APPROVAL

DOUGLAS AIRCRAFT COMPANY, INC — SANTA MONICA, CALIFORNIA

Figure 5.
Assemblies, Terminal and Valve, Cell

CODE IDENT NO.	SIZE	
18355	C	1B10858
SCALE NOTED		SHEET 1 OF 1

REF. DWG. 1B10859 ASSEMBLY

C1642

2.0 TECHNICAL DISCUSSION

2.1 TASK I - Design of Multiplate 5 AH Cell

2.1.1 Grooved Cell Case

The first design concept of a 5 AH multiplate cell consisted of a grooved-wall cell case. In this design the separators were cemented into grooves machined in the side walls of the cases; the electrodes were then inserted and the leads were run through hollow terminals mounted in the cell cover and soldered in place.

A model built in accordance with this design was submitted to environmental testing and satisfactorily proved the test specifications called for in the work statement. These tests, which included vibration, shock and acceleration, are reported in detail in SM-48461-Q1.[1]

2.1.2 Grooved Frame Cell Design

The cell assembly described above was somewhat cumbersome to assemble and could be unreliable on an assembly line basis. A design modification was introduced after approval by the NASA Project Manager. In this design a grooved frame holding separators and electrodes is assembled first. After attaching the electrode tabs to the base of the terminals (previously assembled in the cover), the entire assembly consisting of frame, electrode pack, and attached cover is inserted into a smooth wall cell case. The cover is then assembled to the cell case which is provided with a recess to locate the cover and to provide a sealing area.

To keep the cell size as close as possible to conventional 5 AH cells, the dimensions of all components were held to a minimum. The dimensions of the cell components are shown in Figures 1 through 5.

The assembly of the cell was made as follows:

(a) Cell Case: The case walls and the bottom are sealed together on a mandrel. The case material used is PPO. Chloroform or a 10 to 20% PPO solution in chloroform was used as the cement. The case assembly is dried at room temperature under light pressure. After air drying, the assembly is baked in an oven at 60 - 70°C to eliminate solvent traces.

The assembled case is then tested for gas leakage at five psi pressure and for KOH leakage by filling the case with 45% KOH and letting it stand for 24 hours at room temperature and for 24 hours at 100°C. The case assembly is accepted for use if it passes these tests without evidence of leakage.

(b) Frame: The frame consists of three parts — two sides and one bottom. The three parts have aligned grooves. The separators are assembled by cementing them into the frame grooves.

The spacings between separators are then checked using feeler gages to insure ease of electrode insertion.

(c) Cover: The terminals are assembled to the cover using Shell Resin 901/B-3 which has proven to be leak proof at temperatures ranging from -30°C to 150°C.

(d) Plates: The electrode plates are fabricated with the connector tabs cut to length and punched at the proper location for attachment to the terminal base.

Positive plates are prepared from Handy and Harmon Silpowder 130. Negative plates are prepared from a mixture of 98% ZnO and 2% HgO and are wrapped in KT paper approximately 20 mils thick.

(e) Assembly: The plates are then slipped into their respective cavities. The positive plates are wrapped in a "U" of absorbent material that serves as an electrolyte retainer.

The punched connector tabs are attached to the cell terminals. Finally, the entire assembly is inserted in the case and the cover is sealed in place.

(f) Valve: Presently a valve is used as a means of sealing the cell temporarily until more data are obtained on gassing characteristics and the magnitude of pressure build up which will be encountered during operation at 25° and 100°C.

2.1.3 Molded Cases

To expedite the work on this contract, a survey of existing molds available from several battery manufacturers was made. A case has been selected that very closely approximates the dimensions of the fabricated cases described above. One hundred of these cell cases molded from PPO have been ordered. Delivery is expected about 1 March 1966.

2.2 TASK II - Fabrication, Testing and Evaluation of Cell Components

2.2.1 Electrodes

Electrode specifications have been established covering fabrication procedures, dimensions and weights of active materials.

The current collector used for both positive and negative electrode plates is silver expanded metal, 3 AG 10 - 3/0 to which a 1/4" x .006" silver strip is attached by spotwelding.

The electrode weight is controlled to $\pm 2.5\%$.

During the first quarter, the electrode formulations were:

Positive: 50% Ag powder, 50% monovalent Ag oxide

Negative: 98% zinc oxide, 2% HgO

KT paper pressed on both sides of the electrodes was changed. Based on test results obtained during the first quarter, the positive formulations were changed to Handy-Harmon Silpowder 130 sintered at 650°C for three minutes after electrode pressing. The negative electrode formulation has not been changed.

2.2.1.1 Case Configuration

Two unit cell case configurations have been used in this program for the evaluation of electrodes and inorganic separators. The first design consisted of a two-compartment cell containing one inorganic separator, one positive and one negative electrode. Figure 6 shows this design. This test cell design is designated as "ESC-0000".

The new test cell for electrode and separator testing is shown in Figure 7. This cell consists of a grooved three-sided frame that can hold either one or two inorganic separators so that cells can be tested having either one positive electrode and one negative electrode or one positive and two negative electrodes. This approach is used in guiding the electrochemical development of the electrodes toward the multiplate configuration.

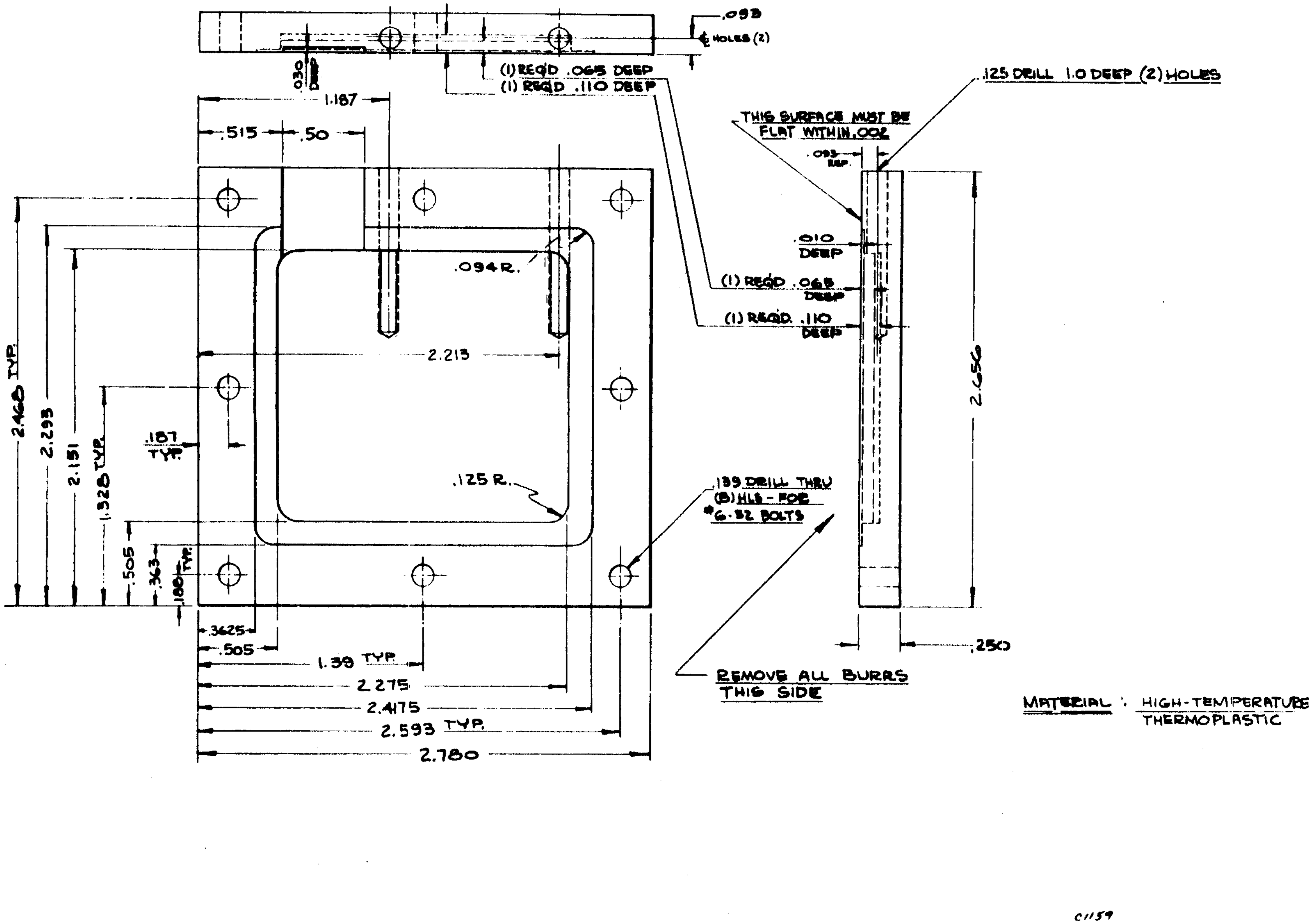
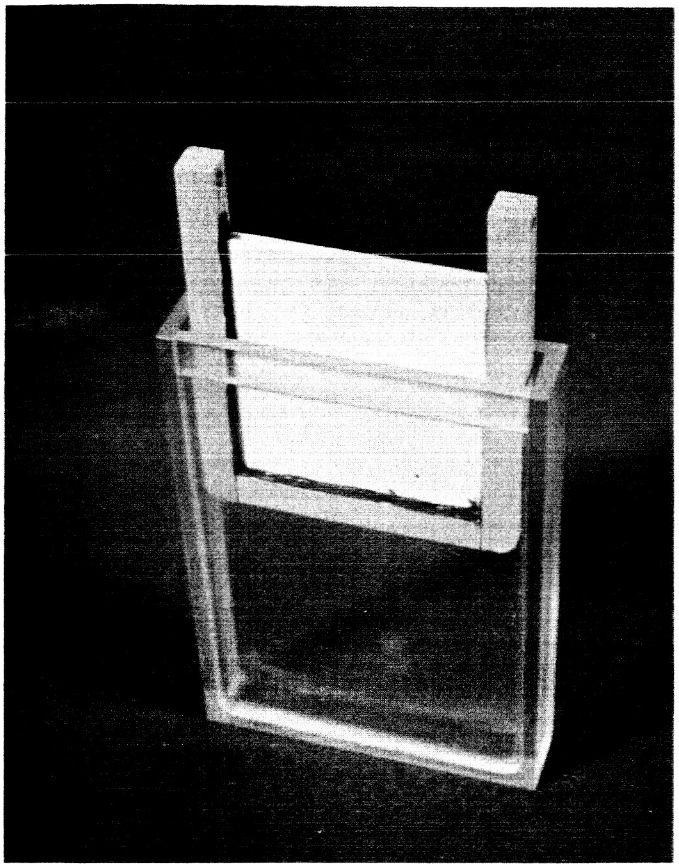


Figure 6. Case, Test Cell, 177091-01-BE (ESC Type)



C/4/0

Figure 7. Single Electrode Test Case With 2-Separator Grooved Frame (ESC-B Type)

Cell cases are being fabricated in Lucite and polystyrene for tests at 25°C and PPO for tests at 100°C.

2.2.1.2 Testing

The principal test for determining electrode characteristics is ability to cycle. In accordance with the work statement, cycle testing is performed on each electrode configuration and formulation at room temperature and at 100°C on a 30-minute discharge, 60-minute charge test schedule using 140 cycles as the cut-off criterion. Electrode effectiveness is first determined by cycling at a discharge current density of 20 ma/cm^2 and then raised until the maximum operating discharge current density is established.

The cycle test data on the single tests cells presented in the first quarterly progress report[1] indicated that the 140-cycle cutoff requirement specified in the work statement was readily obtained on cells at both 100°C and 25°C at a discharge current density of 20 ma/cm^2 and a charge current density of 11 ma/cm^2. When the discharge current density was increased to 27 ma/cm^2, no test cells reached the 140 cycle cutoff due to the higher charge rate required. It was, therefore, decided to establish two experimental cycle test regimes, one at a discharge current density of 20 ma/cm^2 with a charge current density of 11 ma/cm^2, and the other at 40 ma/cm^2 discharge current density and a 22 ma/cm^2 charge current density to permit evaluation of electrodes in order to determine the maximum current density for practical cell operation at 25°C and 100°C.

Table I summarizes the cycle test results for a number of two-plate test cells cycled at 100°C and 25°C at discharge current densities of 20 ma/cm^2 and 40 ma/cm^2. At room temperature four cells completed the required 140 cycles on a 30-minute discharge and 60-minute charge cycle test schedule at 20 ma/cm^2 discharge current density and an 11 ma/cm^2 charge current density. These were Test Cell Nos. ESC-0112, ESC-0113, ESC-0121, and ESC-0127. During assembly of Test Cell No. ESC-0114 the separator was apparently broken, as was evidenced when the cell was disassembled following the completion of 23 cycles. Test Cell Nos. ESC-0128 and ESC-0137 also completed 140 cycles on the same cycle test schedule at

TABLE I

CYCLE TEST RESULTS
ELECTRODE TEST CELL CONFIGURATION - ONE POSITIVE AND ONE NEGATIVE

NOTE: Cycle Test Regime: 30-Minute Discharge and 60-Minute Charge

Test Cell No.	Cycle Test Temp.	Current Density Discharge (ma/cm^2)	Current Density Charge (ma/cm^2)	Number of Cycles Successfully Completed	Remarks
ESC-0122	100°C	20	11	114	Zinc Electrode
ESC-0123				54	
ESC-0124				78	
ESC-0126				120	
ESC-0128				140	Note 1
ESC-0135				101	Zinc Electrode
ESC-0137				140	Note 1
ESC-0130	100°C	40	22	88	Zinc Electrode
ESC-0131				93	
ESC-0132				134	
ESC-0133				60	
ESC-0134				100	
ESC-0136				96	
ESC-0138				100	
ESC-0141				66	
ESC-0112	25°C	20	11	140	Note 1
ESC-0113				140	
ESC-0114				23	Separator Broken during Assembly
ESC-0121				140	Note 1
ESC-0127				140	
ESC-0116	25°C	40	22	46	Zinc Electrode
ESC-0117				21	Cause of Failure Unknown
ESC-0118				140	Note 1
ESC-0119				72	Zinc Electrode

TABLE I (continued)

CYCLE TEST RESULTS

ELECTRODE TEST CELL CONFIGURATION - ONE POSITIVE AND ONE NEGATIVE

Test Cell No.	Cycle Test Temp.	Current Density Discharge (ma/cm^2)	Current Density Charge (ma/cm^2)	Number of Cycles Successfully Completed	Remarks
ESC-0120	25°C	40	22	45	Zinc Electrode
ESC-0140				140	Note 1
ESC-0142				140	Note 1
ESC-0143				79	Zinc Electrode

Note 1. Cell successfully completed the cycling requirement of the contract, however, the cell is capable of continued cycling.

100°C. Three additional test cells (ESC-0122, ESC-0126 and ESC-0135) had completed more than 100 cycles at 100°C and 20 ma/cm^2 discharge current density when they lost capacity due to the depletion of active material in the zinc electrode.

Several test cells (ESC-0118, ESC-0140 and ESC-0142) cycled at 25°C at a discharge current density of 40 ma/cm^2 and a charge current density of 22 ma/cm^2 on a 30-minute discharge and a 60-minute charge test schedule completed the required 140 cycles. Test Cell No. ESC-0132 completed 134 cycles and Test Cell Nos. ESC-0135 and ESC-0138 successfully completed 100 cycles at 100°C and 40 ma/cm^2 discharge current density.

A number of two-plate test cells cycled at 100°C and 25°C at discharge current densities of 40 ma/cm^2 and at 20 ma/cm^2 did not complete 140 cycles. Test Cell No. ESC-0132 completed 134 cycles and Test Cell Nos. ESC-0134 and ESC-0138 completed 100 cycles at 100°C and 40 ma/cm^2 discharge current density.

These cells were tested in half-case assemblies in which electrode cavities were deeper than the thickness of the positive and negative plates. Because of the excessive space available around the zinc electrode, slumping and expansion of the electrode resulted during cycling. This expansion exerted pressure on the separator causing it to bow because of the space available behind it in the positive compartment. The problem appears to be overcome in the new three-electrode test cases where the positive electrode is held firmly between the two separators with one zinc electrode on either side. Because all components fit together in a tight pack, lateral movement is precluded and zinc electrode changes should be minimized.

Recent experimental results show that the cycle performance of the new three-plate cell design is considerably better than that of the former two-plate cell design previously described in the monthly progress report for September 1965.(2) The cell identification number for the three-plate test cell is "B", i.e., ESC-B-000.

Table II is a summary of the three-plate test cells being cycled at 100°C and 25°C on a 1/2-hour discharge and a 1-hour

TABLE II

CYCLE TEST RESULTS

ELECTRODE TEST CELL CONFIGURATION - ONE POSITIVE AND TWO NEGATIVES

NOTE: Cycle Test Regime: 30-Minute Discharge and 60-Minute Charge

Test Cell No.	Cycle Test Temp.	Current Density Discharge (ma/cm^2)	Current Density Charge (ma/cm^2)	Number of Cycles Successfully Completed	Remarks
ESB-B-157	100°C	20	12	140	Note 1 (Table I)
ESC-B-158				140	
ESC-B-159				140	
ESC-B-160				140	
ESC-B-166				28	Silver Electrode Swelled and cracked separator
ESC-B-167				30	Loose Electrode/Separator frame
ESC-B-168	100°C	30	20	50	Test in Progress
ESC-B-169				43	
ESC-B-170				37	
ESC-B-171				21	
ESC-B-175				18	
ESC-B-176				18	
ESC-B-177				12	Cell Case Bulged Due to accidental overheating
ESC-B-181				28	Test in Progress
ESC-B-184				35	
ESC-B-185				34	
ESC-B-186				69	
ESC-B-187				34	
ESC-B-139	25°C	20	12	140	Note (Table I)
ESC-B-144					
ESC-B-145					
ESC-B-146					
ESC-B-147					
ESC-B-148					
ESC-B-149					
ESC-B-150					
ESC-B-151					
ESC-B-152					

TABLE II (continued)

CYCLE TEST RESULTS
ELECTRODE TEST CELL CONFIGURATION - ONE POSITIVE AND TWO NEGATIVES

Test Cell No.	Cycle Test Temp.	Current Density Discharge (ma/cm^2)	Current Density Charge (ma/cm^2)	Number of Cycles Successfully Completed	Remarks
ESC-B-165	25°C	30	20	97	Test in Progress
ESC-B-172				22	Cell Dried Out Completely
ESC-B-173				51	Possible Cell Short
ESC-B-174				52	Test in Progress
ESC-B-178				70	
ESC-B-179				79	
ESC-B-180				67	
ESC-B-153	25°C	40	28	24	Cell reversed*
ESC-B-154				69	Broken lead to zinc electrode
ESC-B-155				121	Test in Progress
ESC-B-156				83	Cell reversed*
ESC-B-161				66	Test in Progress
ESC-B-163				34	Test in Progress

*cycle test timer failure

charge schedule at discharge current densities of 20 ma/cm^2 and 40 ma/cm^2. Ten of these test cells have completed the required 140 cycles at 25°C and 20 ma/cm^2 discharge current density. Several of these new test cells were permitted to continue cycling and have run for more than 400 cycles this far and continue under test. Test Cell Nos. ESC-B-147, ESC-B-149, and ESC-B-152 have cycled at 25°C and at a discharge current density of 20 ma/cm^2 (equivalent to 35% depth of discharge based on rated capacity) and continue under test. Test Cell No. ESC-B-139 has completed 700 cycles to the present time under the same test conditions. These cells were assembled in molded C-11 polystyrene cell cases purchased from a battery manufacturer. A Lucite shim was used in these cell assemblies to provide electrode cavities of the same dimensions as used in the Lucite and PPO cell cases fabricated in the machine shop. Test Cell No. ESC-B-139 is assembled in a Lucite cell case.

Test Cell Nos. ESC-B-157, ESC-B-158, ESC-B-159, and ESC-B-160 have completed the required 140 cycles at 100°C and 20 ma/cm^2 discharge current density indicating that the three-plate test cell design can successfully meet the 140 cycle requirement at both 100°C and 25°C at a discharge current density of 20 ma/cm^2 and a charge current density of 12 ma/cm^2.

Based upon the successful attainment of 140 cycles at both 100°C and 25°C at 20 ma/cm^2 discharge current density and 12 ma/cm^2 charge current density, another group of test cells was placed on cycle test at 25° and 100°C at 30 ma/cm^2 discharge current density and a 20 ma/cm^2 charge current density. These cycle tests are continuing and typical performance data are shown in Table II. Concurrent cycle testing is also in progress at 40 ma/cm^2 discharge current density.

Several of the new test cells are being discharged through a fixed resistance. This method provides a more realistic procedure under flight conditions than constant current operation. The fixed resistance discharge method will be used during the remainder of the program when single test cells are cycled.

Table III shows electrode construction and formulation for all test cells of the new configuration (one positive and two

TABLE III

ELECTRODE TEST CELL CONSTRUCTION - ONE POSITIVE AND TWO NEGATIVES

		Positive Formulation		Negative Formulation					
						KT Paper			
Test Cell Number	Case Material	100% Ag	50-50 Ag/Ag_2O	ZnO %	HgO %	U-Wrap	10% Fibres	Other	Comments
ESC-B-144	Lucite	x		98	2	x			Collector (All Cells): Ag EXMET 3Ag-10-3/0
ESC-B-145		x		98	2			Pellon U-Wrap	
ESC-B-146		x		98	2	x			
ESC-B-147		x		98	2	x			
ESC-B-148		x		98	2	x			
ESC-B-149		x		98	2	x			Lead (All Cells): Ag tab 1/4" x .006" Tab spot welded to grid for all electrodes
ESC-B-150		x		98	2	x			
ESC-B-151		x		98	2	x			
ESC-B-152		x		98	2	x			
ESC-B-153		x		98	2	x			
ESC-B-154	Lucite	x		98	2	x			
ESC-B-155	PPO	x		98	2	x			
ESC-B-156		x		98	2	x			
ESC-B-157		x		98	2	x			
ESC-B-158		x		98	2	x			
ESC-B-159		x		98	2	x			
ESC-B-160		x		98	2	x			
ESC-B-161		x		98	2	x			
ESC-B-162		x		98	2	x			
ESC-B-163		x		98	2	x			
ESC-B-164		x		98	2	x			
ESC-B-165		x		98	2			Asbestos U-Wrap	
ESC-B-166		x		98	2	x			
ESC-B-167		x		98	2	x			
ESC-B-168		x		98	2	x			
ESC-B-169		x		98	2	x			
ESC-B-170		x		98	2	x			
ESC-B-171	PPO	x		98	2	x			
ESC-B-172	Lucite	x		98	2		x		
ESC-B-173		x		98	2		x		
ESC-B-174	Lucite	x		98	2		x		
ESC-B-175	PPO	x		98	2		x		
ESC-B-176		x		98	2		x		
ESC-B-177	PPO	x		98	2		x		
ESC-B-178	Lucite	x		98	2		x		
ESC-B-179	Lucite	x		98	2		x		
ESC-B-180	PPO	x		98	2		x		
ESC-B-181		x		98	2		x		
ESC-B-182		x		98	2		x		
ESC-B-183		x		98	2		x		
ESC-B-184		x		98	2	x			
ESC-B-185		x		98	2	x			
ESC-B-186		x		98	2	x			
ESC-B-187	PPO	x		98	2	x			

negative electrodes and two inorganic separators). The positive electrode formulation was identical for all cells but several different methods were used in applying the network support material in the negative electrode. The electrode current collectors and tabs were of the same design in all cells.

Preliminary cycle testing of multiplate cells for component evaluation continued throughout the report period. Multiplate cell No. MC-2 completed 1,165 cycles at 25°C on a 1/2-hour discharge and 1/2-hour charge cycle test schedule. Prior to placing on cycle, this cell was formed by charging to 2.1 volts and discharging at constant current (1.0 ampere) to 1.0 volt cut-off. At this rate the cell had an initial capacity of 13.0 ampere hours. Following a full recharge, the cell was cycled under the following regimes to obtain charge and discharge rate data:

Discharge	Charge	Discharge	Charge	Total Cycles
4.0 Amps.	2.8 Amps.	1/2 hr.	1 hr.	17 cycles
2.8 Amps.	1.6 Amps.	1/2 hr.	1 hr.	10 cycles
1.0 Amps.	1.6 Amps.	1/2 hr.	1/2 hr.	652 cycles
1.1 Amps.	2.0 Amps.	1/2 hr.	1/2 hr.	513 cycles

Figure 8 shows the 28th and 338th cycles for this multiplate cell. At cycle 338 there was a degradation of the monovalent silver oxide plateau voltage probably caused by geometric changes in the zinc electrode, resulting in reduced area of the zinc electrode which increased current density.

Multiplate Cell No. MC-3 was placed on a 30-minute discharge, 60-minute charge cycle test schedule at 25°C at 2.8 amperes and 2.0 amperes respectively. This cell completed 427 cycles when it was disassembled for analysis.

2.2.2 Inorganic Separator

Inorganic separators have been fabricated during this reporting period as required for component evaluation and cell testing. Process and quality control tests indicate a consistent high level of uniformity and quality.

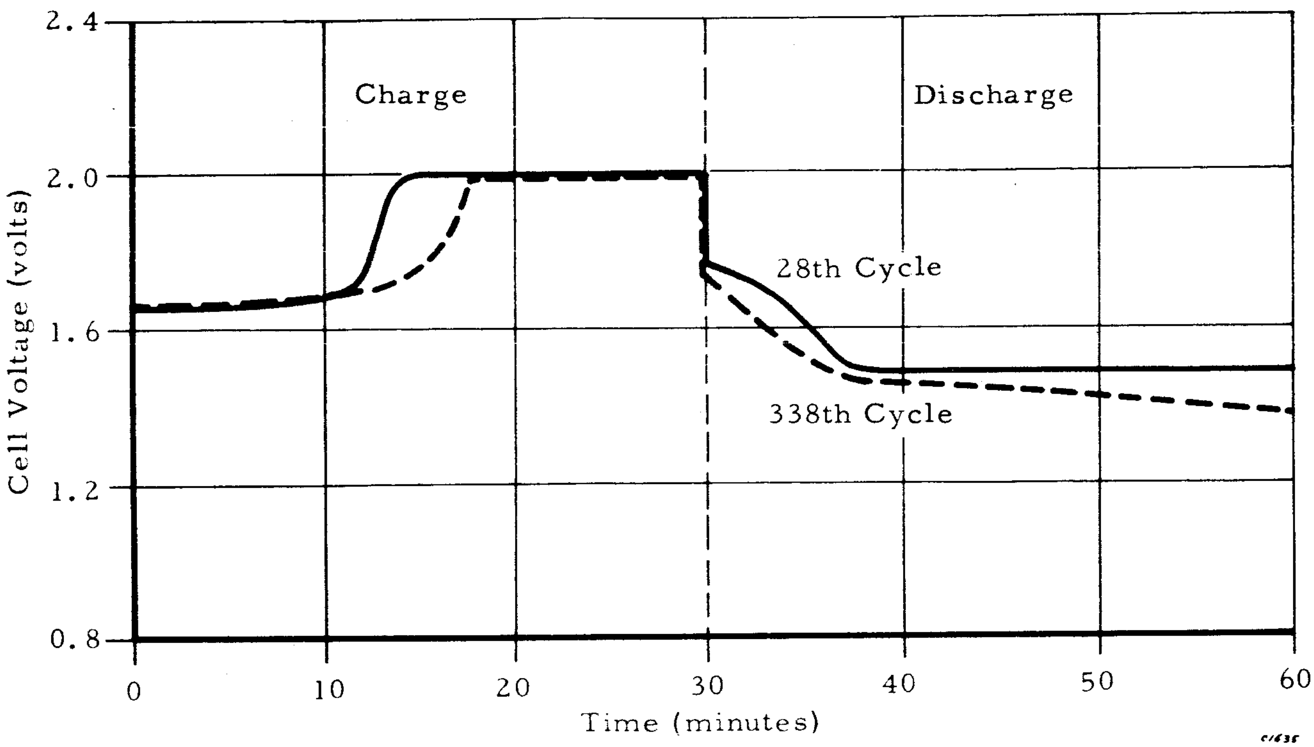


Figure 8. Charge-Discharge Curves Multiplate Silver-Zinc Test Cell Number MC-2 Cycling at Room Temperature at a Test Regime of 30-Minute Discharge and 30-Minute Charge.

2.2.3 Cases

The statement of work requires that a physical property evaluation of cell structural materials be performed in order to select materials having satisfactory strength and rigidity. The following two materials were tested and evaluated.

Material	Manufacturer
Polyphenylene Oxide (PPO)	General Electric Company
Acetal Copolymer (Celcon)	Celanese Plastics Company

The material tests performed included:

(a) modulus of rupture

(b) tensile strength

(c) modulus of elasticity

(d) dimensional compatibility

(e) thermal degradation

One thousand hour KOH-high temperature compatibility tests on PPO and Celcon case materials were completed during the reporting period. These tests involved the complete submergence of test samples of PPO and Celcon (shown in Figure 9) in 35% KOH which were sealed in stainless steel pressure vessels. The sealed pressure vessels were heated to $100^{\circ}C$ $\pm 2^{\circ}C$ for 1000 hours. Figure 10 shows one of the stainless steel pressure vessels used in these tests. At each 100-hour interval, the test samples were removed from the container, washed, dried, and inspected for evidence of degradation, and then returned to the pressure vessel for continuation of the test. The 100-hour inspections showed no visible degradation of either PPO or Celcon after a total of 1000 hours of exposure. Test samples were photographed prior to the start of the tests (Figure 9) and again after completion of the tests (Figure 11). The only visible change which occurred as a result of the test was a change in color of the Celcon test specimens from white to beige where the test specimen protruded from the KOH solution. This discoloration can be noted in Figure 11 at the right ends of the two top Celcon test specimens. The PPO test specimens were unchanged in color as a result of the test.

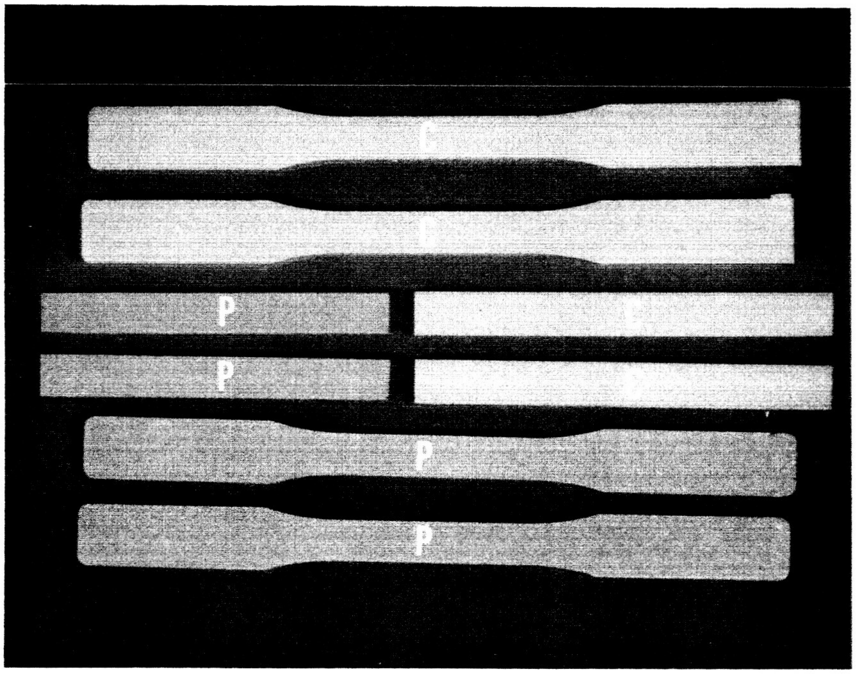


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Figure 9. Test Samples of Celcon and PPO Prior to the 1000-hour KOH Compatibility Test

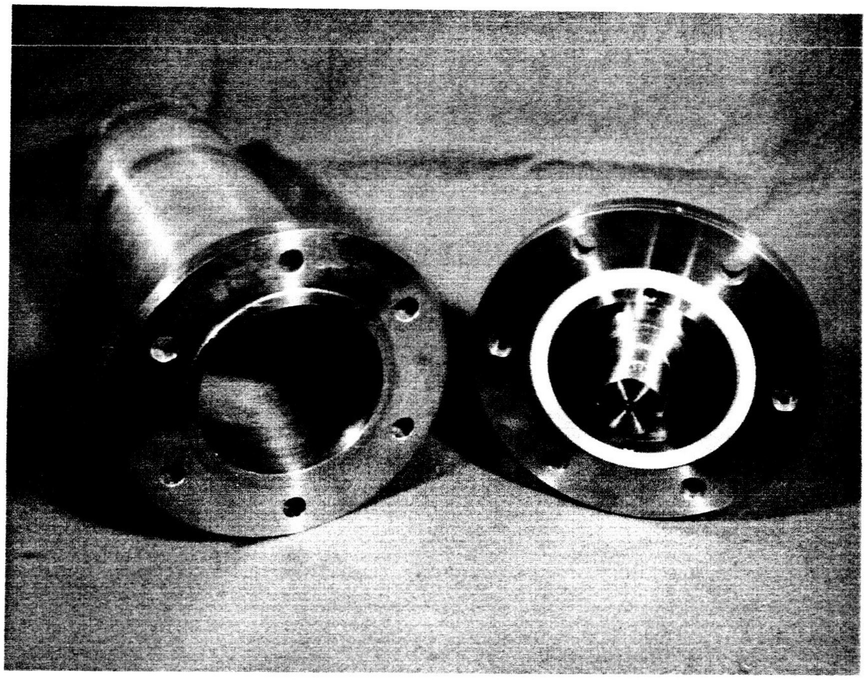

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Figure 10. Stainless Steel Pressure Vessel

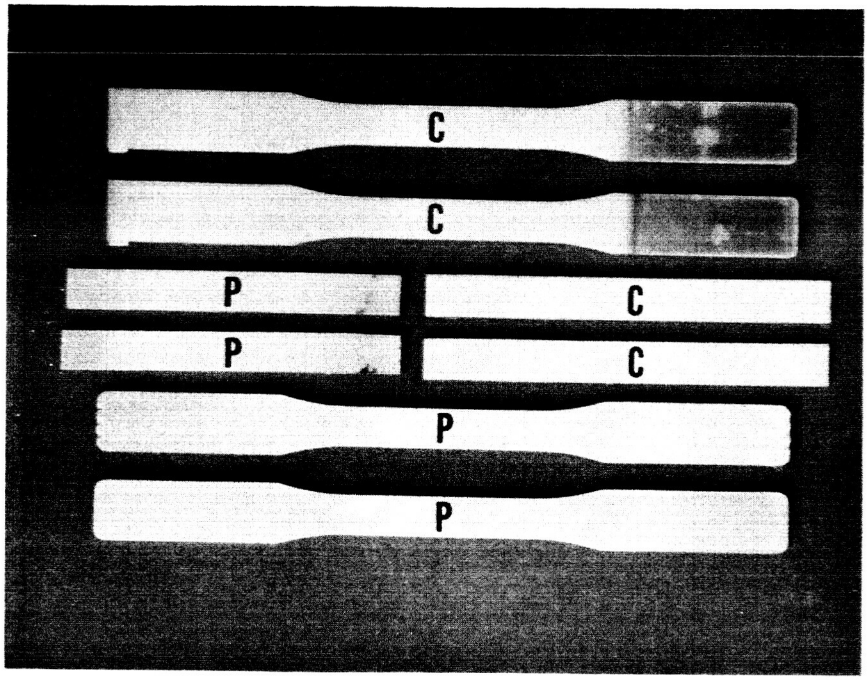

Figure 11. Test Samples of Celcon and P P O Following the 1000-hour K O H Compatibility Test

Test results on the PPO and Celcon test specimens after 1000 hours exposure to KOH at 100°C are shown below:

1. Tensile strength and modulus of elasticity after exposure; tested and computed in accordance with ASTM D-638-64T.

Sample No.	Material	Ultimate Tensile (psi)	Average (psi)	Modulus of Elasticity	Average (psi)
1	Celcon	9,540		3.36×10^5	
2	Celcon	9,370		8.28×10^5	
3	Celcon	9,510	9430	5.17×10^5	5.17×10^5
4	Celcon	9,480		4.18×10^5	
5	Celcon	9,310		5.22×10^5	
6	Celcon	9,360		4.82×10^5	
7	PPO	10,600		5.05×10^5	
8	PPO	10,600		6.01×10^5	
9	PPO	10,600	10600	5.43×10^5	5.64×10^5
10	PPO	10,700		6.08×10^5	
11	PPO	10,700		6.02×10^5	
12	PPO	10,600		5.26×10^5	

2. Modulus of rupture after exposure; tested and computed per ASTM D-790-63.

Sample No.	Material	Modulus of Rupture (psi)	Average (psi)
1	Celcon	11,240	
2	Celcon	10,900	11,000
3	Celcon	10,900	
4	PPO	14,690	
5	PPO	15,210	15,000
6	PPO	15,100	

3. Dimensional stability and weight change after exposure. For this test the thickness and width were measured with a dial thickness gauge, to the nearest 0.001 inch, at several points along each sample before exposure. Following exposure, measurements were taken at identical points and compared with the initial values. In a like manner the length of each sample was measured between

two specific points before and after exposure. For the weight change the samples were weighed on an analytical balance to four decimal places before and after exposure.

Material	Celcon	PPO
Average thickness change	0	0
Average width change	-0.16%	-0.05%
Average length change	-0.24%	0
Average weight change	-0.15%	-0.01%

4. Appearance after exposure: The samples after exposure were compared with unexposed samples for color. The Celcon had changed in color from a milk white to a light blue-gray in the area of total immersion in the solution. In the area where the tensile tabs were out of the solution in the vapor phase the color had changed to a dark beige. The color of both areas was the same in the interior of the sample as on the surface. After testing, the tensile bars turned blue in the areas of strain. The gloss of the Celcon resin was unchanged by exposure. The PPO was essentially unchanged in color and gloss after exposure.

5. Summary of test results:

Material	Test	Manufacturer's Data	Initial Astropower Laboratory (Results)	Final Astropower Laboratory (Results)
Celcon	Ultimate Tensile	8,800 psi	8,470 psi	9,430 psi
Celcon	Modulus of Elasticity	No value given	3.69×10^5 psi	5.17×10^5 psi
Celcon	Modulus of Rupture*	13,000 psi	12,000 psi	11,000 psi
PPO	Ultimate Tensile	9,000 - 10,000 psi	9,460 psi	10,600 psi
PPO	Modulus of Elasticity	$3.5 - 3.8 \times 10^5$ psi	3.4×10^5 psi	3.4×10^5 psi
PPO	Modulus of Rupture*	14,000 - 15,000 psi	13,600 psi	15,000 psi

*The modulus of rupture is a measurement of the load per unit area, placed in the center of a beam of test material, required to develop five (5) percent strain in the outer fiber.

One task to be performed later in the program involves the thermal cycling of activated multiplate cells at 145°C ±2°C for three 36-hour soak periods. It was decided to submit test samples of PPO and Celcon to this test while submerged in 45% KOH during the evaluation of case materials in order to select a case material which would also satisfactorily meet this requirement. Due to an error in calibration of the temperature recorder, a higher temperature was obtained for two and one-half of the 36 hour periods of test than planned. As a result, the test samples of PPO and Celcon were heated to 193°C for 93 hours plus 15 hours at 145°C. The PPO samples were unaffected by this test but the Celcon samples melted into a lump. An additional test on new Celcon test samples was conducted by heating the submerged samples in 45% KOH for 6 hours at 145°C $\pm$2°C. These samples also melted into a lump during this test. Based on these tests it was determined that PPO was a satisfactory case material and that Celcon was unsuitable for use in this program.

The test results on the PPO test specimens are shown below:

1. Tensile strength and modulus of elasticity after exposure to 45% KOH (tested in accordance with ASTM D-638-64T).

Sample No.	Material	Ultimate Tensile (psi)	Average (psi)	Modulus of Elasticity	Average (psi)
1	PPO	11,250		3.47×10^5	
2	PPO	11,250	11,260	3.58×10^5	3.49×10^5
3	PPO	11,340		3.51×10^5	
4	PPO	11,190		3.40×10^5	

2. Modulus of rupture, after exposure (tested in accordance with ASTM D-790-63).

Sample No.	Material	Modulus of Rupture (psi)	Average (psi)
1	PPO	14,590	
2	PPO	14,980	14,900
3	PPO	14,720	
4	PPO	15,330	

3. Dimensional Stability: For this test the thickness and width were measured with a dial thickness gauge in the nearest 0.001 inch, at several points along each sample, before exposure. Following exposure, measurements were taken at identical points and compared with the initial values. In a like manner the length of each sample was measured between two specific points before and after exposure.

Average thickness change	+1.6%
Average width change	+0.3%
Average length change	-1.4%

4. Weight Change: The samples were weighed on an analytical balance to four decimal places before and after exposure.

Average weight change	-0.01%

Based upon preliminary sealing tests, it also appears that PPO can be sealed by solvent bonding, thereby effecting a satisfactory seal between cell cover and case.

At the suggestion of the NASA Project Officer, molded polystyrene cell cases and covers of the approximate 5 AH cell size have been purchased. These cell cases and covers are being used for evaluations at 25°C. Molded 5 AH cell cases and covers made from PPO have also been ordered for tests at 100°C and higher.

2.2.4 Terminals

As reported in the first Quarterly Report,[1] the terminal seal design successfully passed thermal cycling, pressure and KOH leakage tests as required by the work statement.

After testing in accordance with these specifications, no trace of alkalinity was detected at the external end of the terminal during the test period and as long as 24 hours later. The terminal was disassembled for examination and no attack could be detected on any material used or on the entire configuration. The attachment of electrode tabs to the base of the terminal was modified subsequently for ease of assembly, as shown in Figure 4.

2.2.5 Connections and Current Collectors

The electrode tab (current collector) connection to the cell terminal consists of two basic designs, (1) silver tabs spot-welded or hot-forged to silver expanded metal grids cut to size, and (2) silver expanded metal grid in which a solid silver tab is an integral part of the grid. A KOH soak test was performed, in compliance with the statement of work, in which two electrode current collector grids of the first design described above were completely submerged in 35% KOH in a stainless steel vessel shown in Figure 10 and the vessel maintained at $150^{\circ}C \pm 2^{\circ}C$ for 168 hours. At the completion of the 168-hour soak test, the current collector grids were removed from the sealed vessel, washed, dried, and inspected under a microscope for corrosion. No evidence of corrosion was noted. Figure 12 is a photograph of the current collector grids taken after the 168-hour soak test. A 30 ampere current (dc) was then passed through each grid for a period of one minute with one current lead connected to the end of the silver tab and the other current lead attached to the bottom of the silver grid. No evidence of resistance, corrosion or discoloration was detected. Finally an attempt was made to mechanically pull the tab away from the grid at the welded joint by holding the grid in a vise and manually pulling the tab. The tab could not be pulled loose from the grid indicating satisfactory weld strength. Assemblies were also tested to determine the force required to pull the tab away from the collector. It was determined that when 10 pounds force was applied, the Exmet grid pulled apart. The welded joint between tab and grid was unaffected by the test showing that the weld is stronger than the grid material itself.

Because the second type is an integral part of the current collector-connector assembly, it was unnecessary to test this design for corrosion resistance. Current tests were conducted which indicated that the physical design is adequate for the proposed application and capable of operation continuously at 30 amperes.

2.3 Summary of Results and Conclusions

Several prototype multiplate 5 ampere-hour cells were fabricated throughout the past quarter. The cell design used was the modified groove-type, which was approved by the NASA/Lewis Project Officer early in this quarter. This design emphasizes the requirements of the work statement, such as ease of assembly, positive closures, parts reproducibility, adequate closures, and filling means. Cell assembly and detail parts drawings were completed on this modified groove-type multiplate cell design. One preliminary multiplate cell of this design completed 1165 cycles at $25^{\circ}C$ on a 30-minute discharge and 30-minute charge test schedule.

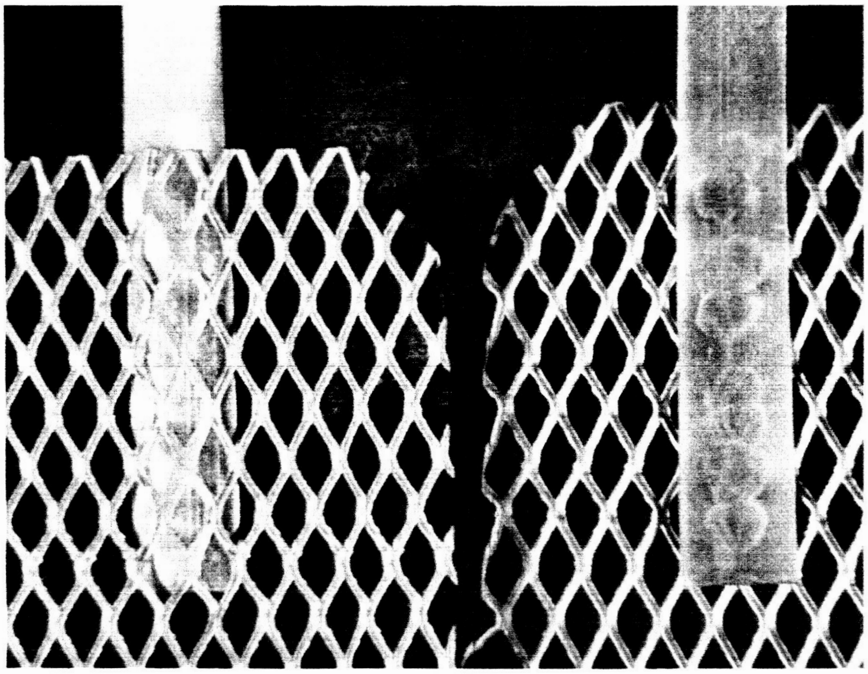

Figure 12. Silver Electrode Current Collector Grids Following Exposure to 150°C for 168 Hours in 35% KOH

Molded cell cases made of polyphenylene oxide (PPO) have been ordered for use in this program for both 100°C and 25°C temperature tests. The molded case approximates very closely the dimensions of the fabricated 5 AH multiplate cell design.

Final test data and analyses were obtained on the two candidate cell case and cover materials — PPO and Celcon — which were subjected to the 1000-hour KOH-high temperature soak tests. As a result of these tests, it was apparent that PPO can be used as the material for the multiplate cells to be built in Task III. Celcon can not withstand the 145°C specified in the statement of work. The mechanical properties of PPO remained unchanged as a result of the exposure.

Astroset Type 5-036-011 inorganic separators were fabricated and incorporated into test cells and prototype multiplate test cells during the quarter. Separator quality and uniformity have continually been improved as a result of strict quality control measures which have been implemented throughout this program.

Cycle testing of unit electrode test cells containing Astroset Type 5-036-011 inorganic separators was continued at 100°C and 25°C for the purpose of evaluating electrode formulations and configurations. Many electrode test cells successfully completed the required 140 cycles in compliance with the statement of work at 100°C and 25°C at a discharge current density of 20 ma/cm^2. A new three-plate unit cell design is being cycle tested at room temperature and 100°C at discharge current densities of 20 ma/cm^2 and 30 ma/cm^2.

Based on the promising experimental test cell results obtained during the past quarter, the emphasis of the program has been shifted from component testing to multiplate cell fabrication, testing and evaluation. Although the component work will be continued at a reduced level of effort, multiplate cell work will be carried out based on the best components developed thus far. These cells will be constructed with PPO cases, and electrodes which have given satisfactory results. Astroset Type 5-036-011 inorganic separators will be used in the construction of cells.

3.0 WORK PLANNED

3.1 TASK I - Design of a Multiplate 5 AH Cell

3.1.1 Modify terminal base for tab attachment and complete drawings.

3.1.2 Modify separator dimensions to fit frame grooves more deeply.

3.1.3 Design and fabricate new separator die.

3.2 TASK II - Fabrication, Testing and Evaluation of Cell Components (Single Electrode Cell)

3.2.1 Electrodes

3.2.1.1 Fabricate electrodes, assemble single electrode cells and test them mainly at current densities higher than 20 ma/cm^2 at 25^oC and 100^oC, in order to establish the practical limit capable of reaching 140 cycles reliably and without undue capacity loss at 100^oC.

3.2.1.2 Test single electrode cells at 25^oC and 100^oC for gassing rates on stand and on cycling.

3.2.2 Separators

Fabricate separators from Astroset Type 5-036-011 to new dimensions as required for component and cell testing.

3.2.3 Case

3.2.3.1 Assemble single electrode cell cases, cement and test for leakage.

3.2.3.2 Adapt cover and stainless steel tubing for 100^oC operation and minimization of water evaporation.

3.2.4 Terminal

Work on terminal seal completed, but base to be modified: See next paragraph.

3.2.5 Connections

The base of the terminal was redesigned in order to simplify the tab attachment from plates to terminals during cell assembly.

Terminals with new bases are being fabricated and will be used on the new test cells.

3.2.6 Electrolyte

Study effects of 30, 35, 40, 45%, KOH concentration on cell cycle life and drain rate.

Four cells will be built and tested at various load regimes for 3 cycles, then cycled at the 1 hour-rate of discharge at 25°C for life.

3.2.7 Multiplate Cells

1. Order 100 PPO cell cases from molder for multiplate cells. The case was selected for its dimensions approximating our design very closely except for height. The cases will be cut down to the proper height and pressure-tested at 125°C and 30 psi.

2. Case-to-cover bond will be pressure tested to determine rupture pressure.

3. Multiplate cells will be built and tested at 25° and 100°C.

4.0 PERSONNEL

As of 30 January 1966, a total of 6270 engineering hours have been expended on this project. The personnel who worked on this program and the approximate percentage of their time devoted to this project are as follows:

Dr. C. Berger	No direct charge
F. C. Arrance	100%
A. Himy	50%
A. D. Taylor	50%
A. G. Rosa	100%
Q. H. McKenna	100%
R. K. Greve	20%
M. P. Kilgore	60%
J. D. Armantrout	20%

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2. "Program to Develop an Inorganic Separator for a High Temperature Silver-Zinc Battery," Contract NAS-37639, Report SM-48461-M2 September (1965).

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Attn: J. F. Donahue

Gulton Industries
Alkaline Battery Division
Metuchen, New Jersey
Attn: Dr. Robert Shain

Hughes Research Laboratories Corporation
Malibu, California
Attn: T. M. Hahn

Inland Testing Laboratories
Dayton, Ohio
Attn: W. Ingling

ITT Federal Laboratories
500 Washington Avenue
Nutley 10, New Jersey
Attn: Dr. P. E. Lighty

Livingston Electronic Corporation
Route 309
Montgomeryville, Pennsylvania
Attn: William F. Meyers

Lockheed Missiles and Space Company
Department 65-82
Palo Alto, California
Attn: Larry E. Nelson

Mallory Battery Company
60 Elm Street
North Terrytown, New York
Attn: R. R. Clune

Midwest Research Institute
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Attn: Dr. B. W. Beadle

North American Aviation, Incorporated
12214 Lakewood Boulevard
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Attn: Burton M. Otzinger

P. R. Mallory & Company, Incorporated
Northwest Industrial Park
Burlington, Massachusetts
Attn: Dr. R. Selim

Power Sources Division
Whittaker Corporation
P. O. Box 337
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Attn: Mr. John Rhyne

Radio Corporation of America
Astro Division
Heightstown, New Jersey
Attn: Seymour Winkler

Space Technology Laboratories, Incorporated
One Space Park
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Southwest Research Institute
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Texas Instruments, Incorporated
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Attn: Dr. I. Trachtenberg

Sonotone Corporation
Saw Mill River Road
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Attn: A. Mundel

Thomas A. Edison Research Laboratory
McGraw Edison Company
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Union Carbide Corporation
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P. O. Box 6056
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Westinghouse Electric Corporation
Research & Development Center
Churchill Borough
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Attn: Dr. A. Langer

Western Company
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Attn: R. T. Fiske

Yardney Electric Corporation
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Attn: Dr. Paul Howard

Aerospace Corporation
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